भारतीय मानक **IS 5280 : 2023**

Indian Standard

फेनिट्रोथियोन तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

Fenitrothion Technical — Specification

(First Revision)

ICS 65.100.10

भारतीय मानक ब्यूरो
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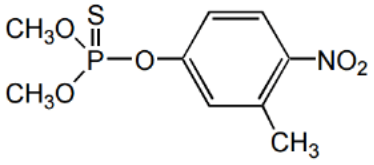
FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Divisional Council.

Fenitrothion, technical is employed in the preparation of insecticidal formulations.

Fenitrothion is the common name accepted by the International Organization for Standardization for the pesticide chemical containing *O,O*-dimethyl *O*-(3-methyl-4-nitrophenyl) phosphorothioate (*see* Note) as its active ingredient. The empirical and structural formulae and the molecular weight of the compound are as indicated below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_9H_{12}O_5PSN$		277.24

NOTE — The other chemical name accepted by the International Organization for Standardization for this pesticide is dimethyl 3-methyl-4-nltrophenyl pholphorothionate.

This standard was first published in 1969 and subsequently amended to modify Table 1 of the standard through one amendment.

In year 2007 vide S.O. 706 (E) dated 3 May 2007, the use of fenitrothion was banned in agriculture except for locust control in scheduled desert area and for public health by Government of India.

In this revision, the spectroscopic method for determination of fenitrothion content has been incorporated. Also, the standard has been brought out in the latest style and format of the Indian Standards and suitable modification in the scope informing the users regarding application of the fenitrothion for locust control in scheduled desert area and for public health only. It also incorporates one amendment issued to this standard.

The composition of the Committee responsible for the formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed, or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

FENITROTHION TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of test for fenitrothion, technical employed in the preparation of insecticidal formulations.

1.2 Fenitrothion, technical may be used for locust control in scheduled desert area and for public health only.

2 REFERENCES

The standards, given below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards.

IS No.	*Title*
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 2362 : 1993	Determination of water by Karl Fischer method — Test method (*second revision*)
IS 6940 : 1982	Methods of test for pesticides and their formulations (*first revision)*
IS 8190 (Part 2) : 1988	Requirements for packaging of pesticides: Part 2 Liquid pesticides (*second revision*)
IS 10946 : 1996	Methods of sampling for technical grade pesticides (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be amber coloured, slightly oily liquid, free from extraneous impurities or modifying agents and it shall be insoluble in water.

3.2 The material shall comply with the requirements given in Table 1.

Table 1 Requirements for Fenitrothion, Technical
(*Clause* 3.2)

Sl No.	Characteristics	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Fenitrothion content, percent by mass, *Min*	95	Annex A
ii)	Matter insoluble in acetone, percent by mass, *Max*	0.5	IS 6940
iii)	Moisture content, percent by mass, *Max*	0.5	IS 2362
iv)	Acidity (as H_2SO_4), percent by mass, *Max*	0.5	IS 6940
v)	Specific gravity at 27° /27 °C	1.310 – 1.320	IS 6940

4 PACKING

The material shall be packed as per requirements given in IS 8190 (Part 2)

5 MARKING

5.1 The containers shall be securely closed and shall bear legibly and indelibly the following information in addition to any other information as required under the *Insecticides Act*, 1968 and Rules framed there under:

a) Name of the material;

b) Name and address of the manufacturer;

c) Batch number;

d) Date of manufacture;

e) Date of expiry;

f) Net quantity;

g) Nominal fenitrothion content, percent (*m/m*);

h) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and

j) Any other information required under the *Legal Metrology (Packaged Commodities) Rules*, 2011.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn according to IS 10946.

7 TESTS

7.1 Tests shall be carried out by the methods referred to in co1 **4** of the Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE —'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A
[*Table* 1, *Sl. No.* (i)]

DETERMINATION OF FENITROTHION CONTENT

A-1 GENERAL

A-1.1 Two methods, namely, titration method (**A-2**) and spectroscopic method (**A-3**) may be used for determination of fenitrothion content. The spectroscopic method shall be used as a referee method.

A-2 TITRATION METHOD

A-2.1 Principle

A-2.1.1 The sample is dissolved in ether and the most likely impurity, 4-nitro-m- cresol. is extracted with mild alkali. The ether layer is treated with zinc and an acetic acid- hydrochloric acid mixture, and the amino groups are titrated with standard sodium nitrite solution.

A-2.2 Reagents

A-2.2.1 *Acetic Acid - Hydrochloric Acid Mixture*

Mix 9 volumes of glacial acetic acid with one volume of concentrated hydrochloric acid.

A-2.2.2 *Standard Potassium Nitrite Solution*

0.1 M, standardized against sulphanilic acid. Dissolve 6·90 g of sodium nitrite in distilled water and make up to one litre with distilled water. Standardize as follows:

Weigh accurately 0.40-0.45 g of anhydrous sulphanilic acid (analytical grade) into a 400 ml tall-form beaker. Add 80 ml of distilled water, 10 ml of concentrated hydrochloric acid, 30 ml of glacial acetic acid and 5 g of sodium (or potassium) bromide. Cool the mixture to 0° to 10 °C by the addition of clean, shaved ice and stir mechanically. Titrate at 0° to 10 °C with the 0.1 M sodium nitrite as rapidly as the spot test permits. Near the end-point, add the sodium nitrite in four-drop portions.

Normality of 0.1 M sodium nitrite = $\frac{a \times 5.774}{b}$

where

a = weight (in g) of sulphanilic acid used, and
b = volume (in ml) of 0.1 M sodium nitrite required.

A-2.3 Procedure

A-2.3.1 *Extraction of Free 4-nitro-m-cresol*

Weigh accurately about 1 g of the sample and transfer the same in a 200 ml separating funnel containing 100 ml of ether. Extract the ether solution four times with 20 ml portions of chilled 1 percent (*w*/*v*) sodium carbonate solution and discard the aqueous layers. The extraction should be done as quickly as possible to prevent decomposition of the active ingredient.

A-2.3.2 *Determination of O,O-dimethyl O-(3-Methyl-4-Nitrophenyl) phosphorothioate in Ether Layer*

Transfer the ether layer quantitatively to a 400 ml beaker, using small portions of ether. Add 100 ml of the acetic acid-hydrochloric acid mixture and 10 g of iron-free zinc dust. Cover the beaker with a watch-glass and heat gently on a steam-bath until most of the ether has evaporated and the solution is colourless. Add 25 ml of concentrated hydrochloric acid and 75 ml of distilled water to complete the solution of the zinc dust. Cool, wash down the beaker and watch-glass with distilled water, and add five grams of sodium (or potassium) bromide. Cool the mixture to 0° to 10 °C by the addition of clean, shaved ice and stir mechanically. Titrate at 0° to 10°C with the standardized 0.1 M sodium nitrite as rapidly as the spot test permits. Near the end-point, add the sodium nitrite in four-drop portions.

A-2.3.3 *Spot Test*

Dip a glass rod into the solution to be tested and then touch the rod quickly to a piece of potassium iodide-starch paper. The end-point is reached when an intense blue black colour appears immediately and may be obtained repeatedly during a one-minute period without further addition of sodium nitrite.

A-2.4 Calculation

O,O-dimethyl *O*-(3-Methyl-4-Nitrophenyl) phosphorothioate content, percent by mass = $\frac{27.72\ W\ N\ f}{w}$

where

W = volume (in ml) of 0.1 M sodium nitrite required,
N = normality of 0.1 M sodium nitrite,
f = correction factor T/A,
w = weight (in g) of sample, and

where

T = quantity (in ml) of 0.1 M sodium nitrite calculated for the nitro-group determination of one gram of a mono-nitro reference material, and A = quantity (in ml) of 0.1 M sodium nitrite used in the nitro-group determination of one gram of a recrystallized sample of the same substance, following the method described above and using the same reagents.

NOTE — The correction factor *f* is intended to allow for errors due to impurities in the reagents as well as those inherent in the method itself or in its application by a given laboratory. Its value shall lie within the range 0.98 to 1.02.

A-3 SPECTROSCOPIC METHOD

A-3.1 Principle

Sodium salt of 4-nitro-m- cresol is obtained by the alkaline hydrolysis of fenitrothion which is determined colorimetrically at 410 nm by spectroscopy and is then converted into fenitrothion. A free 4-nitro-m- cresol present in the sample is determined and corrected after applying the conversion factor to obtain actual content of fenitrothion.

A-3.2 Apparatus

A-3.2.1 *Standard Flasks* – 100 ml, 250 ml, 500 ml, and 1 000 ml Capacity.

A-3.2.2 *Pipettes* – 20 ml, 25 ml, and 50 ml.

A-3.2.3 *Beakers*

A-3.2.4 *Round Bottom Flask (GG Joint)* – 250 ml Capacity.

A-3.2.5 *Conical Flasks* – 250 ml Capacity with Standard Joints.

A-3.2.6 *Separating Funnel* – 250 ml Capacity.

A-3.2.7 *UV Visible Spectrophotometer*.

A-3.3 Reagents

A-3.3.1 *Methanol*

A-3.3.2 *Diethyl Ether*

A-3.3.3 *Sodium Hydroxide* – 1 N

A-3.3.4 *Chilled Sodium Carbonate* – 1 %

A-3.3.5 *4-Nitro-m- Cresol* – AR grade

A-3.4 Procedure

A-3.4.1 *Determination of Total 4-Nitro-m- Cresol*

Weigh accurately about 0.5 g of 4-nitro-m- cresol AR grade in to 250 ml capacity gg flask. To it add 50 ml of methanol and 25 ml of 1 N NaOH solution. Heat the mixture under reflux condenser for 1 h. Cool for 15 minutes and wash the condenser with distilled water, cool to room temperature and transfer the contents quantitatively into a 1 000 ml standard flask and make up to the mark with distilled water. Shake well to get a uniform solution. Call this solution ‘A’.

A-3.4.2 Transfer 5 ml of solution ‘A’ in to a 250 ml standard flask and make up the volume with distilled water. Shake well to get a uniform solution. Call this solution ‘B’.
Measure the absorbance of solution ‘B’ at 410 nm against distilled water as blank.

A-3.5 Preparation of Test Solution

A-3.5.1 *Preparation of Fenitrothion Sample Solution*

Weigh accurately an amount of sample equivalent to 0.85 g of active ingredient into a 250 ml gg flask and proceed exactly as described in **A-3.4**.

A-3.6 Determination of Free 4-Nitro-m-Cresol

A-3.6.1 Weigh accurately about 0.05 g of 4-nitro-m-cresol AR grade in to a 250 ml standard flask and add 1% sodium carbonate solution, mix well and make up to the mark with 1% sodium carbonate solution, shake well to homogenize the solution. Measure the absorbance of this solution at 410 nm against 1% sodium carbonate solution as blank.

A-3.6.2 Weigh accurately an amount of sample equivalent to 0.08 g of fenitrothion active ingredient into a beaker and transfer in to a 250 ml separating funnel containing 50 ml solvent ether quantitatively with about another 50 ml of solvent ether. Extract the free 4-nitro-m- cresol with 20 ml of 1% chilled sodium carbonate solution. Continue the extraction till the aqueous sodium carbonate solution (aqueous phase) is colourless. Depending on the volume of extracted carbonate washings transfer it to a 250 ml or 500 ml or 1 000 ml standard flask quantitatively and make up with 1% sodium carbonate solution. Shake well to get a uniform solution. Measure the absorbance of this solution at 410 nm against 1% sodium carbonate solution as blank.

A-3.7 Calculation

A-3.7.1 Total 4-nitro-m-cresol content (X) = $\frac{A_2 \times M_1 \times P}{A_1 \times M_2}$

where

A_1 = absorbance of 4-nitro-m- cresol,
A_2 = absorbance of sample,
M_1 = mass of 4-nitro-m- cresol AR grade,
M_2 = mass of test sample, and
P = purity of 4-nitro-m- cresol AR grade.

A-3.7.2 Free 4-nitro-m- cresol (Y) = $\frac{A_4 \times M_3 \times P \times dilution\ factor}{A_3 \times M_4}$

where

A_3= absorbance of 4-nitro-m- cresol,
A_4 = absorbance of sample,
M_3 = mass of 4-nitro-m- cresol AR grade,
M_4 = mass of test sample, and
P = purity of 4-nitro-m- cresol AR grade.
Dilution Factor: for 250 ml = 1
500 ml = 2
1 000 ml = 4

A-3.7.3 Fenitrothion content, percent by mass = (X – Y) × 1.81 (conv. factor)

ANNEX B
(*Foreword*)

COMMITTEE COMPOSITION

Pesticides Sectional Committee, FAD 1

Organization	*Representative(s)*
Directorate of Plant Protection Quarantine and Storage, Faridabad	DR RAVI PRAKASH (***Chairperson***)
All India Biotech Association, New Delhi	SHRI SAURABH SINGHAL SHRI SHAH JI DHAR (*Alternate*)
Central Insecticide Board and Registration Committee, Faridabad	SECRETARY DR VANDANA SETH (*Alternate*)
Central Insecticide Laboratory, Faridabad	DR ARCHANA SINHA SHRI SUBHASH CHAUDHARY (*Alternate*)
Consumer Guidance Society of India, Mumbai	SHRI SITARAM DIXIT DR M. S. KAMATH (*Alternate*)
Crop Care Federation of India, New Delhi	DR J. C. MAJUMDAR
Crop Life India, New Delhi	SHRI ASITAVA SEN MS NIRUPAMA SHARMA (*Alternate*)
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Indian Agricultural Research Institute, New Delhi	DIRECTOR
Indian Institute of Packaging, Mumbai	DR TANWEER ALAM
Indian Pest Control Association, New Delhi	SHRI UDAYAN GHOSH
Institute of Pesticide Formulation Technology, Gurgaon	DR M. VAIRAMANI
Ministry of Agriculture, Department of Agriculture, Chennai	JOINT DIRECTOR OF AGRICULTURE (RES) DEPUTY DIRECTOR LAB (*Alternate*)
National Centre for Integrated Pest Management, New Delhi	DR SUMITRA ARORA
National Institute of Plant Health Management, Hyderabad	DR MAHESH SAINI MS T. SRIDEVI (*Alternate*)
Pesticide Manufactures and Formulators Association of India (PMFAI), Mumbai	DR ARCHANA SRIVASTAVA DR UDAY KUMAR (*Alternate*)
Regional Pesticides Testing Laboratory, Chandigarh	SHRI V. VASU

Organization	*Representative(s)*
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In Personal Capacity (*263, Sector 28, Faridabad – 121008, Haryana*)	SHRI VIPIN SAINI
BIS Directorate General	SHRIMATI SUNEETI TOTEJA, SCIENTIST 'E' AND HEAD (FOOD AND AGRICULTURE) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
SHRI KULDEEP MITTAL
SCIENTIST 'B'/ASSISTANT DIRECTOR
(FOOD AND AGRICULTURE), BIS

Bureau of Indian Standards

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Review of Indian Standards

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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